

Final:

DEFENSE NATIONAL STOCKPILE CENTER PROGRAMMATIC SAMPLING AND ANALYSIS PLAN

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SECTION 1

PROGRAM DESCRIPTION

1.1 DNSC MISSION

The Defense National Stockpile Center (DNSC) operates depots across the country for the purpose of stockpiling resources deemed to be critical to national security. Each of the manned Depots and their locations are listed on Table 1.1 below. The primary materials stockpiled are: raw ore resources, processed ore and metals storage in ingot form, and containerized materials stored in warehouses. Most of the metals and raw and processed ores are stored outdoors in piles that are not covered. The depots have been operable since the 1940s. The need for the stockpiled minerals has diminished due to the end of the cold war, and the DNSC is in the process of selling the materials and consolidating the remaining inventory at specific depots such that some depots can be closed.

TABLE 1.1

DNSC DEPOT LOCATIONS

| DEPOT NAME | LOCATION | STATUS |
|----------------------|-------------------------------|--------|
| Baton Rouge Depot | Baton Rouge, Louisiana | Active |
| Binghamton Depot | Binghamton, New York | Active |
| Clearfield Depot | Clearfield, Utah | Active |
| Curtis Bay Depot | Curtis Bay, Maryland | Active |
| Gadsen Depot | Gadsen, Alabama | Active |
| Hammond Depot | Hammond, Indiana | Active |
| Mechanicsburg Depot | Mechanicsburg, Pennsylvania | Active |
| New Haven Depot | New Haven, Indiana | Active |
| Point Pleasant Depot | Point Pleasant, West Virginia | Active |
| Scotia Depot | Scotia, New York | Active |
| Sharonville Depot | Sharonville, Ohio | Active |
| Somerville Depot | Hillsborough, New Jersey | Active |
| Warren Depot | Warren, Ohio | Active |

1.2 PROGRAM BACKGROUND

As part of the process of consolidating and closing some of the depots, site characterization assessments are being conducted to determine the impacts that the depots have had on the environment over their periods of operation. Parsons will be conducting those site characterization projects under contract to the United States Army Engineering and Support Center, Huntsville (USAESCH). The purpose of this Programmatic Sampling and Analysis Plan (PSAP) is to provide the basic sample collection, methodologies, analytical techniques, quality

assurance/quality control (QA/QC) requirements, data validation, and analytical reporting procedures that may be applied to the site characterization projects. A site/project-specific addendum to this PSAP will be prepared for each site characterization to identify the site history, results from any previous investigations (if applicable), the site-specific sampling rationale, all applicable regulations/standards, nonmeasurement data acquisition, the project schedule, hypotheses being tested, the number of samples being collected, the locations of the samples, and the specific analytes of interest. The combination of the project-specific addendum and this PSAP will provide all the information needed to collect and analyze the samples in accordance with applicable USAESCH requirements.

SECTION 2

PROGRAM ORGANIZATION AND RESPONSIBILITIES

Four organizations will be involved in this program of site characterization projects at various depots; they are as follows:

- DNSC - The DNSC operates the depots and is responsible for providing background information and site access to the field teams. The primary DNSC personnel responsible for the site characterization program are F. Kevin Reilly and Stephen Surface, located in Fort Belvoir, Virginia.
- USAESCH - the USAESCH is responsible to the DNSC for management of the site characterization program, evaluating quality assurance data and reviewing data quality reports provided by Parsons. The USAESCH Program Manager is Bruce Railey, located in Huntsville, Alabama.
- Parsons Engineering Science (Parsons) – Parsons, under contract DACA87-02-D-0005 with USAESCH, has been awarded a delivery order (0005) to execute the site characterization program at various DNSC depots. The Parsons Contract Manager is Todd Heino, P.E., located in Boston, Massachusetts. The Parsons Delivery Order Manager and Point of Contact (POC) is George Moreau, located in Syracuse, New York. Mr. Moreau will be responsible for evaluating the data and compiling the investigation reports. The Parsons Quality Control Manager will be Maryanne Kosciwicz. Mrs. Kosciwicz will also be responsible for data assessment organization and validation.
- General Engineering Laboratories, Inc. (GEL) - GEL is the analytical laboratory that, under a subcontract to Parsons, is responsible to perform the chemical analyses for each of the site characterization projects. The GEL Program Manager is Valerie Davis, located in Charleston, South Carolina. Valerie will be the POC for GEL and will report to George Moreau of Parsons. Laboratory-specific information for GEL is provided throughout this document and in Appendix A which includes a listing of method detection limits and various state and USACE certification paperwork. GEL shall not subcontract any sample analyses without prior documented approval from Parsons and the USACE Contracting Officer. If GEL is unable to perform any analyses, an alternate laboratory will be utilized with approval from USAESCH. All non-GEL laboratory-specific information will be included in the site-specific project addenda.

SECTION 3

PROGRAMMATIC QUALITY ASSURANCE OBJECTIVES

3.1 DATA QUALITY OBJECTIVES

Data quality objectives (DQOs) are qualitative and quantitative statements that specify the quality and the level of the data required to support the decision-making processes during each site characterization project. Guidance for the DQO development process is contained in *EM 200-1-2, Technical Project Planning Process* (USACE, 1998), *Guidance for Performing Site Inspections Under CERCLA* (USEPA, 1992), *Guidance For Preparation of Sampling and Analysis Plans* (USACE, 2001) and *Data Quality Objectives Process* (USEPA, 2000). The data collection objectives, the data uses, and the appropriate analytical data quality level(s) for each sample medium are identified in this section.

3.1.1 Program Objective

The overall objective of the site characterization program is to assess the impact a depot may have had on the environment as a result of its operation by DNSC. The objective of this PSAP is to provide guidance such that the site characterization data are of known and documented quality and useable for their intended purposes.

Site characterization samples will be collected from locations where environmental impacts are suspected to exist, such as within the “footprints” of stockpiled commodities and along surface water drainage and groundwater flow paths. The presence of environmental impacts will be assessed by comparing sample concentrations to current Federal or State regulatory criteria and site-specific background concentrations.

The methods anticipated to be used to obtain project data are listed on Table 3.1.

3.1.2 Analytical Data Quality Levels

The analytical data quality levels for the data acquisition program are defined as follows:

- Screening analyses produce real-time results that are typically used for field monitoring and laboratory confirmation sample selection. Field analysis is not anticipated for this program. If site-specific screening analyses are required, details will be included in the applicable project specific work plan addendum.
- Definitive analyses of parameters will use the most current promulgated methods for laboratory analyses. The methods presented in Table 3.1 are anticipated to be used by GEL to obtain project data. This data quality level provides compound-specific results with established quantitation limits, accuracy and precision.

3.1.3 Analytical Practical Quantitation Limits

Table A.1 of Appendix A presents the “standard” practical quantitation limits (PQLs) for all laboratory analytical methods, by compound and sample matrix. If a State, Federal or other applicable regulatory criteria requires that lower quantitation limits be achieved, those variances will be addressed in the appropriate project-specific work plan. The laboratory-reported method detection limits (MDLs) are specified by the analytical methodology listed in USEPA SW-846. The laboratory will report sample results greater than the MDL but less than the PQL, which in the analyst’s professional judgment are believed to be reliably detected, as estimated values. Any additional parameters required for a particular site will be included in the applicable project specific work plan addendum.

3.1.4 Soil, Groundwater, Surface Water and Sediment Samples

The objective of soil, groundwater, surface water, and sediment sample collection is to assess whether hazardous substances are present at concentrations above regulatory criteria and site-specific background, which may indicate environmental impacts have occurred. The project-specific addendum will describe the use of any screening analyses that may be performed in the field (i.e. for volatile organic compounds) to help select samples for laboratory analyses.

3.2 QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA

Data required to satisfy the program and project-specific objectives must meet minimum quality assurance criteria. This section describes data quality assurance objectives in terms of precision, accuracy, representativeness, completeness, and comparability (PARCC) for both laboratory and field programs.

3.2.1 Laboratory and Field DQO and PARCC Parameters

Control or recognition of sampling, analytical, and data reduction errors is important in analyzing the data and in preparing the final site characterization report. DQOs for laboratory analytical data are usually expressed in terms of PARCC parameters. Definitions and descriptions of these PARCC parameters are presented in the following subsections. The calculations for precision, accuracy and completeness are described in Section 7. The field data quality objectives can be divided into two major segments: those pertaining to field screening and field instruments, and those related to sample selection, collection, and shipping. Descriptions of each PARCC parameter are presented below. Field screening measurements that may be made include pH, temperature, turbidity and conductivity measurements, headspace analysis by PID or Flame Ionization Detector (FID), and radiological surveys. Section 4 provides approved procedures for performing the field screening activities.

3.2.1.1 Precision

3.2.1.1.1 Precision is the measure of the variability of individual sample measurements. Laboratory precision will be inferred through the use of laboratory duplicate samples – matrix spike/matrix spike duplicate (MS/MSD) samples, laboratory control sample/laboratory control sample duplicate (LCS/LCSD) and sample duplicate, and field duplicate samples. Frequency of QC sampling is discussed in Subsection 3.5. The laboratory-specific precision values for

MS/MSDs and LCS/LCSDs are listed on Table 3.2 for each applicable SW846 (or project-specific) method.

3.2.1.1.2 Sampling and shipping precision will be controlled by adhering to the approved procedures presented in Sections 4 and 5. Sampling and shipping precision for the quantitative samples will be inferred through the results of the field duplicates sent to the laboratory for analysis. Precision of the qualitative samples will be inferred through the results of the field analysis duplicates. The frequency of the field duplicates is discussed in Subsection 3.5. The precision value for field duplicates shall not exceed 50% relative percent difference (RPD).

3.2.1.2 Accuracy

3.2.1.2.1 Accuracy is a measure of the analytical bias. Bias is defined as the difference between the actual sample analyte value and the measured/reported laboratory sample analyte value. The exact analytical bias cannot be determined since the actual sample analyte value is not known. Examination of laboratory blank analytical data, laboratory matrix spiked analytical data (MS/MSDs, LCS/LCSDs), and surrogate compound recoveries (as required by method) allow any bias introduced by laboratory sample handling and procedures to be assessed. The laboratory-specific accuracy values for MS/MSDs and LCS/LCSDs are sample matrix-dependant and are specified on Table 3.2 for each applicable (or project-specific) analytical method. The laboratory-specific accuracy values for surrogates are sample matrix-dependant and are specified on Table 3.3 for each applicable (or project-specific) analytical method

3.2.1.2.2 Accuracy for field screening procedures will be controlled through the proper calibration of the instruments, adherence to the operational procedures, and documentation of all observations and readings. Performing all sampling and shipping procedures according to the approved procedures presented in Sections 4 and 5, will help to assure that adequate field accuracy is achieved. The accuracy for the qualitative field samples will be inferred through the results of the calibration procedures as described in the instrument manufacturer's manual and the results of the equipment blanks. Calibration must take place each time the instrument is turned on prior to sample analysis. Equipment blanks will be evaluated to assess the presence or absence of contamination from other samples, sampling procedures, sample containers, or preservatives.

3.2.1.3 Representativeness

3.2.1.3.1 Representativeness is the degree to which a set of data accurately reproduces the characteristics of the population. Analyses performed by the analytical laboratory will follow standard analytical methodologies for data representativeness by the methods presented in Section 6. Instrument performance checks, initial and continuing calibrations, laboratory blanks, internal standards, and sample homogenization are some of the measures evaluated to determine that representativeness has been achieved. The criteria for these measures are defined in the specific analytical method.

3.2.1.3.2 Representativeness for field screening procedures will be controlled through the proper calibration of the instruments, adherence to the operational procedures, documentation of all observations and readings, and proper selection of the measurement point. Due to the

qualitative and general measurements of the field screening analyses, representativeness values are not defined. Representativeness can be assumed for groundwater sampling by purging the aquifer to a point at which indicator parameters stabilize prior to sample collection as described in Section 4. Surface water, soil, and sediment samples will not necessarily be representative of the entire site. Sampling locations may be the areas suspected to have the highest degree of hazardous substances. By following the approved procedures in Section 5, shipping and handling representativeness can be assumed. Due to the qualitative and general measurements of the sampling and shipping procedures, representativeness values are not defined.

3.2.1.4 Completeness

3.2.1.4.1 Completeness is a measure of the amount of data actually collected, analyzed, and validated compared to the amount specified in the sampling plan. Laboratory completeness is based on the number of samples shipped to the laboratory and the number of validated analyses performed. The goal for laboratory completeness will be 90 percent. The overall measure of completeness achieved will be the ratio of the valid results to the total number of results.

3.2.1.4.2 Field completeness is based on the number of samples planned and the number of samples taken. In order to complete the site characterizations, all samples outlined in the site-specific addendum should be collected. The minimum goal for field completeness is 90% for each project. However, field teams should take all measures necessary to complete the sampling program.

3.2.1.5 Comparability

3.2.1.5.1 Comparability expresses the confidence with which one set of data can be compared with another. Comparability can be related to precision and accuracy as these quantities are measures of data reliability. All measurement data generated will be expressed in units as defined by the method to allow comparability of data among sampling locations, as well as between depots, as applicable. The laboratory will maintain a QA/QC program and perform analyses in accordance with applicable USEPA protocols and other applicable standard methods.

3.2.1.5.2 All field measurement data generated will be expressed in units as defined by the method to allow comparability of data among sampling locations, as well as between depots, as applicable. The field team will perform all procedures according to the approved procedures in Sections 4 and 5 to maintain comparability throughout the sampling effort.

3.3 SAMPLE RECEIPT AND HOLDING TIME REQUIREMENTS

3.3.1 Verification / Documentation of Cooler Receipt Condition

A COC record accompanies the sample container from the laboratory to the field where the sample is contained, preserved, and then returned to the laboratory. The laboratory's sample custody program meets the criteria listed below:

- The laboratory has designated a sample custodian who is responsible for maintaining sample custody and for maintaining all associated records documenting sample custody.

- Upon receipt of the samples, the custodian measures and records sample temperature (using the temperature blank) on a cooler receipt form, checks for proper preservation, and checks the original COC documents and compares them with the labeled contents of each sample container for correctness and traceability. The custodian signs the COC record and records the date and time the samples are received. In the event of discrepant documentation or temperature of temp blank outside $4^{\circ}\text{C} + 2^{\circ}\text{C}$, the laboratory immediately contacts the Parsons PM as part of the corrective action process. Sample results for samples in coolers with temperature blanks above 6°C but below 10°C will be qualified during the data validation process. Samples in coolers with temperature blanks above 10°C should be recollected.
- A qualitative assessment of each sample container is performed to note any anomalies, such as broken or leaking containers. This assessment will be recorded as part of incoming COC procedures.
- The samples are stored in a secured refrigerator until analyses begin. Refrigerators will be maintained at $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$, and the temperatures recorded daily.
- A copy of the COC and cooler receipt forms accompanies the laboratory report and becomes a permanent part of the project records.

3.3.2 Corrective Action for Incoming Samples

If any discrepancies are detected during the sample login process at either facility, the sample custodian will immediately notify the Laboratory PM and the Parsons PM.

3.3.3 Holding Times

To maximize representativeness of sample results, all samples will be extracted and/or analyzed within the holding times specified in each method. Tables 3.4 and 3.5 present the maximum holding times allowed for each water and soil method, respectively. State-specific requirements for holding times will be addressed in the project-specific work plan addendum. Extraction or analysis performed after the expiration of the holding time will result in the qualification of the results during the data validation process. Samples that grossly exceed laboratory-holding times for extraction or analysis may be resampled and resubmitted for analysis.

3.4 QA/QC SAMPLE PROGRAM

The analytical data for all samples must be sufficient in both precision and accuracy to identify the compounds present and the respective concentrations. Field duplicates, rinsate blanks, and trip blanks will be collected and submitted to the analytical laboratory for assessing the quality of the sample data. Field duplicate samples will be collected and analyzed to check for sample reproducibility. Rinsate blank samples will be collected and analyzed to check for systematic errors in the sampling and analytical procedures, and ambient site conditions. The Project Chemist, or the Chemist's designee, will review the data to evaluate the precision and accuracy of the data generated. The USAESCH will also evaluate the QA data and review the

data quality reports provided by Parsons. The types of QA/QC samples to be collected for each project are described below, and will be specified in the project-specific work plan.

3.4.1 Trip Blank

3.4.1.1 Trip blanks will be collected and analyzed for volatile organic compounds. The analytical results will serve as a baseline measurement of volatile organic contamination that samples may be exposed to during transport and laboratory storage prior to analysis.

3.4.1.2 Trip blanks originate in the laboratory. They are HPLC-Grade water placed in sample containers, transported to the sample collection site, handled with the samples, and returned to the laboratory with samples of water collected for volatile organic analysis. The trip blank containers are not to be opened in the field.

3.4.1.3 One trip blank will be sent to the laboratory for each day water samples are sent to the laboratory for volatile organic analyses. The trip blank will be stored in the laboratory with the associated samples and will be analyzed by the laboratory for volatile organics.

3.4.2 Equipment Blank

Equipment blank samples are samples of clean analyte-free water passed through and over the sampling equipment. These blanks permit evaluation of equipment decontamination procedures and potential cross-contamination of environmental samples between sampling locations. The number of equipment blanks for each project will vary from site to site and be dictated by state regulations. However, one equipment blank will typically be obtained from each type of sampling tool used to collect environmental samples.

3.4.3 Field Duplicate Samples

Field duplicate samples are samples collected simultaneously from the same media source under identical conditions, then homogenized and split into separate containers. Ten percent of the samples for each matrix will typically be collected in duplicate and submitted for laboratory analysis. Field duplicates will be labeled so that persons performing laboratory analyses cannot distinguish duplicates from other samples.

3.4.4 Matrix Spike / Matrix Spike Duplicate Samples

3.4.4.1 For laboratory analyses, matrix spike and matrix spike duplicate (MS/MSD) samples help assess analytical interferences caused by the sampled matrix. The MS/MSD samples are spiked by the analytical laboratory with known concentrations of specified compounds, and then analyzed. The percent recovery is calculated and is used to evaluate interference effects.

3.4.4.2 One sample for MS and one sample for MSD will be analyzed for every 20 environmental samples of each medium at a site. The samples for MS/MSD may be site-specific and collected immediately after an original sample. In that case, the MS/MSD samples will be labeled with the same identifier as the original; however, the MS/MSD sample labels and chain-of-custody forms will note that the samples are to be used as MS/MSDs. In certain instances, the

MS/MSD analyses may be conducted on a “batch” quality control basis (i.e. in the event there are very few field samples). The project-specific work plan will provide details of the MS/MSD sample collection/selection process.

3.4.5 Laboratory Control Sample / Laboratory Control Sample Duplicate

The laboratory control sample (LCS) is analyte-free water for aqueous analyses or Ottawa sand for soil analyses (except metals where glass beads may be used) spiked with all target analytes for the method. Each analyte in the LCS shall be spiked at a level less than or equal to the midpoint of the calibration curve for each analyte. The LCS shall be carried through the complete sample preparation and analysis procedure. One LCS shall be included in each analytical batch of not more than 20 environmental samples.

3.4.6 QA Split

The QA split is defined as a field duplicate/triplicate that is collected from the same location as the parent sample under identical conditions. The sample is sent to a USACE-designated laboratory for independent analysis. The QA split sample will provide a measure of the representativeness of the sampling procedure. The QA split sample containers will be filled immediately after the field duplicate samples. The required frequency will depend on USACE requests, but in most cases is limited to ten percent of all samples collected per site. The sample ID will be identical to the parent sample with the addition of “QA” to the name.

3.4.7 Temperature Blank

A temperature blank is a container (e.g., 40 mL) of water packaged along with field samples in the shipping cooler that will represent the temperature of the incoming cooler upon receipt at the laboratory. Use of these samples within a shipping container is required and enables the receiving laboratory to assess the temperature of the shipment without disturbing any project field samples. All coolers will contain a minimum of one temperature blank. If cooler temperature is above criteria, the Parsons PM will contact USAESCH.

3.5 LABORATORY QC PROCEDURES

3.5.1 Analytical Sequence QC

Laboratory analyses will be conducted in accordance with the appropriate analytical method sequence requirements. The specific analytical sequence varies for each method, but all require a valid calibration and system (method) blank prior to the analysis of samples.

3.5.2 Batch/Matrix-Specific QC

3.5.2.1 Laboratory QC checks will include reagent or method blanks and surrogate and MS/MSD recoveries. The frequency, acceptance criteria, and corrective action requirements for each laboratory check are specified in the applicable analytical method.

3.5.2.2 Batch-specific QC are those QC procedures applied to an interference-free matrix or a matrix of known composition. Batch-specific QC includes method blanks, standard reference materials, calibration verification standards. They ensure the sampling procedures are

appropriate and the analytical methods are being performed in an in-control mode of operation. However, these QC checks provide no information on how well the method is performing with respect to the project sample matrix. Batch-specific QC will be specified in the applicable analytical method.

3.5.2.3 Method blanks (MBs) are analyzed to assess background interference or contamination that exists in the analytical system that might lead to the reporting of elevated concentration levels or false positive data. The MB is defined as an interference-free blank matrix similar to the sample matrix to which all reagents are added in the same volumes or proportions as used in sample preparation and carried through the complete sample preparation, cleanup, and determinative procedures. The results of the MB analysis are evaluated, in conjunction with other QC information, to determine the acceptability of the data generated for that batch of samples. MB requirements are specified in the analytical method.

3.5.2.4 The analysis of standard reference materials should be included in the laboratory QC program, as required by the analytical method. Standard reference materials are natural standards obtained from a different source than the calibration/spiking standards analyzed as an internal performance evaluation (PE) samples. In addition, the laboratory shall participate in external PE programs.

3.5.2.5 The initial calibration curve shall be verified as accurate with a standard purchased or prepared from an independent source. This initial calibration verification (ICV) involves the analysis of a standard containing all of the target analytes, typically in the middle of the calibration range, each time the initial calibration is performed. The percent recovery of each target analyte in the ICV is determined from the initial calibration and compared with the specifications in each analytical method.

3.5.2.6 Continuing calibration verifications (CCVs) are analyzed to determine whether the analytical system is working properly, and if a new initial calibration (and the reanalysis of sample extracts) is required. Calibration verification differs in concept and practice from continuing calibration. In this latter technique, a standard is analyzed and new response factors are calculated, or a new calibration curve is drawn from the analysis of the continuing calibration standard. The former verifies compliance with the initial calibration curve, but does not overwrite the response factors used for the quantitation, nor allows resloping of the calibration curve. Calibration verification shall be in accordance with the analytical methods, calculating a percent drift when the initial calibration is based on regression analysis, and a percent difference when the initial calibration is determined based upon percent Relative Standard Deviation (RSD) values. CCV typically involves the analysis of a single primary source standard in the middle of the calibration range, between the concentrations of low-level and mid-level calibration standards. The frequencies of the CCV vary between methods but are related to the type of detector used and sample matrices analyzed. This frequency is typically presented within SW-846 methods as at the beginning of the analytical shift/sequence; every 12 hours of analyses or every 10 to 20 samples; and may include at the end of the analytical sequence.

3.5.2.7 Matrix-specific QC are those procedures applied to field samples to provide information on the precision and bias of the analyses on project samples. These procedures include analyses of field samples in association with surrogate compounds and recoveries. Matrix-specific procedures performed on other field samples at the laboratory not associated with the project samples are of no value, for they do not provide information on the matrix under observation.

3.5.2.8 Surrogates are analyzed to assess the ability of the method to successfully recover these specific nontarget analytes from an actual matrix. Surrogates are organic compounds that are similar to the analytes of interest in chemical behavior but are not normally found in environmental samples. Surrogates to be used are identified within the determinative methods. Other compounds may be chosen and used as surrogates, depending on the analysis requirements, whether they are representative of the compounds being analyzed, and whether they cover the chromatographic range of interest. The surrogates will be spiked into all samples and accompanying QC samples requiring gas chromatography (GC), liquid chromatography, or gas chromatography/mass spectrometry (GC/MS) analysis prior to any sample manipulation. As a result, the surrogates are used in much the same way that MSs are used, but cannot replace the function of the MS. The results of the surrogates are evaluated, in conjunction with other QC information, to determine the effect of the matrix on the bias of the individual sample determinations.

TABLE 3.1
SAMPLE ANALYSIS AND EXTRACTION METHODS

| PARAMETER | WATER METHODS | | SOIL METHODS | |
|------------------------------|-------------------------|------------|--------------------------------|------------|
| | ANALYSIS | EXTRACTION | ANALYSIS | EXTRACTION |
| TAL Metals | SW6010B | SW3005A | SW6010B | SW3050B |
| Mercury | SW7470A | | SW7471A | |
| SVOCs | SW8270C | SW3510C | SW8270C | SW3550B |
| VOCs | SW5035A/ SW8260B | | SW5035A/ SW8260B | |
| Pesticides | SW8081A | SW3510C | SW8081A | SW3550B |
| Herbicides | SW8151A | | SW8151A | |
| PCBs | SW8082 | SW3510C | SW8082 | SW3550B |
| Cyanide | SW9012A | SW9010B | SW9012A | SW9010B |
| TCLP Extraction | | SW1311 | | SW1311 |
| Total Organic Carbon | SW9060 Lloyd Kahn | | SW9060 Lloyd Kahn | |
| Total Petroleum Hydrocarbons | SW8015M | SW3510C | SW8015M | SW3550B |
| Thorium | TH-NAS-3004 | | TH-NAS-3004 | |
| Gross Alpha and Beta | SW9310 | | SW9310 | |
| TSS | EPA 160.2 | | NA | |
| BOD-5 Day | EPA 405.1 | | NA | |
| COD | EPA 410.4 | | NA | |
| pH, Corrosivity | EPA 150.1 | | SW9045C | |
| TKN | EPA 351.2 | | EPA 351.2 Mod | |
| Oil & Grease* | EPA 413.1/ EPA 1664A | | NA | |
| Nitrate as N | EPA 300.0 | | SW9056 | |
| Nitrite as N | EPA 300.0 | | SW9056 | |
| Total Organic Nitrogen* | EPA 351.2/ 350.1 | | EPA 351.2 Mod/ 350.1 Mod | |
| Total Dissolved Solids | EPA 160.1 | | NA | |
| Ammonia as N | EPA 350.1 | | EPA 350.1 Mod | |
| Total Hardness | SW6010B | SW3005A | NA | |
| Total Residual Chlorine | EPA 330.5 | | NA | |
| Specific Conductance | EPA 120.1 | | NA | |
| Temperature | EPA 170.1 | | NA | |
| Turbidity | EPA 180.1 | | NA | |
| Ignitability* | | | SW 846 1010 or SW 846 1020A | |
| Hexavalent Chromium | SW 7196A | | SW 7196A | SW 3060 |

* - Specific method to be used will be determined by State regulatory requirements.

TABLE 3.2
Precision and Accuracy Values
for MS/MSDs and LCS/LCSDs

| VOA Solid 8260B/5035 Analyte | LCS and MS Lower Control Limit | LCS and MS Upper Control Limit | LCS and MS Lower ME Limit | LCS and MS Upper ME Limit | MSD %RPD |
|--|-----------------------------------|-----------------------------------|------------------------------|------------------------------|-------------|
| 1,1,1,2-Tetrachloroethane | 75 | 125 | 65 | 135 | 0 - 30 |
| 1,1,1-Trichloroethane | 70 | 135 | 55 | 145 | 0 - 30 |
| 1,1,2,2-Tetrachloroethane | 55 | 130 | 40 | 145 | 0 - 30 |
| 1,1,2-Trichloroethane | 60 | 125 | 50 | 140 | 0 - 30 |
| 1,1-Dichloroethane | 75 | 125 | 65 | 135 | 0 - 30 |
| 1,1-Dichloroethylene* | 65 | 135 | 55 | 150 | 0 - 30 |
| 1,1-Dichloropropene | 70 | 135 | 60 | 145 | 0 - 30 |
| 1,2,3-Trichlorobenzene | 60 | 135 | 50 | 145 | 0 - 30 |
| 1,2,3-Trichloropropane | 65 | 130 | 50 | 140 | 0 - 30 |
| 1,2,4-Trichlorobenzene | 65 | 130 | 55 | 140 | 0 - 30 |
| 1,2,4-Trimethylbenzene | 65 | 135 | 55 | 145 | 0 - 30 |
| 1,2-Dibromo-3-chloropropane | 40 | 135 | 25 | 150 | 0 - 30 |
| 1,2-Dibromoethane (Ethylene Dibromide) | 70 | 125 | 60 | 135 | 0 - 30 |
| 1,2-Dichlorobenzene | 75 | 120 | 65 | 125 | 0 - 30 |
| 1,2-Dichloroethane* | 70 | 135 | 60 | 145 | 0 - 30 |
| 1,2-Dichloropropane | 70 | 120 | 65 | 125 | 0 - 30 |
| 1,3,5-trimethylbenzene | 65 | 135 | 55 | 145 | 0 - 30 |
| 1,3-Dichlorobenzene | 70 | 125 | 65 | 135 | 0 - 30 |
| 1,3-Dichloropropane | 75 | 125 | 70 | 130 | 0 - 30 |
| 1,4-Dichlorobenzene | 70 | 125 | 65 | 135 | 0 - 30 |
| 2,2-Dichloropropane | 65 | 135 | 55 | 145 | 0 - 30 |
| 2-Butanone (MEK)* | 30 | 160 | 10 | 180 | 0 - 30 |
| 2-Chlorotoluene | 70 | 130 | 60 | 140 | 0 - 30 |
| 2-Hexanone | 45 | 145 | 30 | 160 | 0 - 30 |
| 4-Chlorotoluene | 75 | 125 | 65 | 135 | 0 - 30 |
| 4-Methyl-2-pentanone (MIBK) | 45 | 145 | 30 | 165 | 0 - 30 |
| Acetone | 20 | 160 | 10 | 180 | 0 - 30 |
| Benzene* | 75 | 125 | 65 | 135 | 0 - 30 |
| Bromobenzene | 65 | 120 | 55 | 130 | 0 - 30 |
| Bromochloromethane | 70 | 125 | 60 | 135 | 0 - 30 |
| Bromodichloromethane | 70 | 130 | 60 | 135 | 0 - 30 |
| Bromoform | 55 | 135 | 45 | 150 | 0 - 30 |
| Bromomethane (Methyl Bromide) | 30 | 160 | 10 | 180 | 0 - 30 |
| Carbon disulfide | 45 | 160 | 30 | 180 | 0 - 30 |
| Carbon tetrachloride* | 65 | 135 | 55 | 145 | 0 - 30 |
| Chlorobenzene | 75 | 125 | 65 | 130 | 0 - 30 |
| Chlorodibromomethane | 65 | 130 | 55 | 140 | 0 - 30 |
| Chloroethane | 40 | 155 | 20 | 175 | 0 - 30 |
| Chloroform* | 70 | 125 | 65 | 135 | 0 - 30 |
| Chloromethane (Methyl Chloride) | 50 | 130 | 40 | 140 | 0 - 30 |
| cis-1,2-Dichloroethylene | 65 | 125 | 55 | 135 | 0 - 30 |
| cis-1,3-Dichloropropylene | 70 | 125 | 65 | 135 | 0 - 30 |
| Dibromomethane | 75 | 130 | 65 | 135 | 0 - 30 |
| Dichlorodifluoromethane | 35 | 135 | 15 | 155 | 0 - 30 |
| Ethylbenzene | 75 | 125 | 65 | 135 | 0 - 30 |
| Hexachlorobutadiene | 55 | 140 | 40 | 155 | 0 - 30 |
| Isopropylbenzene | 75 | 130 | 70 | 140 | 0 - 30 |
| m,p-Xylene | 80 | 125 | 70 | 135 | 0 - 30 |
| Methyl Tert-butyl Ether (MTBE) | 60 | 125 | 45 | 140 | 0 - 30 |
| Methylene chloride | 55 | 140 | 40 | 155 | 0 - 30 |
| Naphthlaene | 40 | 125 | 25 | 140 | 0 - 30 |
| n-Butylbenzene | 65 | 140 | 50 | 150 | 0 - 30 |
| n-Propylbenzene | 65 | 135 | 50 | 145 | 0 - 30 |
| o-Xylene | 75 | 125 | 70 | 135 | 0 - 30 |
| p-Isopropyltoluene | 75 | 135 | 65 | 140 | 0 - 30 |
| sec-Butylbenzene | 65 | 130 | 50 | 145 | 0 - 30 |
| Styrene | 75 | 125 | 65 | 135 | 0 - 30 |
| tert-Butylbenzene | 65 | 130 | 55 | 145 | 0 - 30 |
| Tetrachloroethene* | 65 | 140 | 55 | 150 | 0 - 30 |
| Toluene* | 70 | 125 | 60 | 135 | 0 - 30 |
| trans-1,2-Dichloroethene | 65 | 135 | 55 | 145 | 0 - 30 |
| trans-1,3-Dichloropropene | 65 | 125 | 55 | 140 | 0 - 30 |
| Trichloroethylene* | 75 | 125 | 70 | 130 | 0 - 30 |
| Trichlorofluoromethane | 25 | 185 | 10 | 215 | 0 - 30 |
| Vinyl Acetate | 64 | 136 | 55 | 145 | 0 - 30 |
| Vinyl chloride* | 60 | 125 | 45 | 140 | 0 - 30 |
| Xylene (Total) | 70 | 130 | 60 | 140 | 0 - 30 |

* TCLP Target Analyte

Table 3.2
Precision and Accuracy Values
for MS/MSDs and LCS/LCSDs

D.2 Marginal Exceedance

DoD will allow a number of sporadic marginal exceedances of the LCS-CLs. The number of exceedances is based on the total number of analytes spiked in the LCS. As the number of analytes in the LCS increases, more marginal exceedances are allowed. The number of allowable marginal exceedances is based on a policy decision that no more than 5% of the total number of analytes spiked in the LCS may exceed the DoD limits. Table D-1 presents the allowable number of marginal exceedances for a given number of analytes in the LCS.

TABLE D-1. NUMBER OF MARGINAL EXCEEDANCES

| Number of Analytes in LCS | Number of Marginal Exceedances of LCS-CLs |
|---------------------------|---|
| >90 | 5 |
| 71–90 | 4 |
| 51–70 | 3 |
| 31–50 | 2 |
| 11–30 | 1 |
| <11 | 0 |

A marginal exceedance is defined as beyond the LCS-CL but still within the marginal exceedance limits (set at 4 standard deviations around the mean). This outside boundary prevents a grossly out-of-control LCS from passing.

DoD requires that the marginal exceedances be sporadic (i.e., random). If the same analyte exceeds the LCS-CL repeatedly (e.g., 2 out of 3 consecutive LCS), that is an indication that the problem is systemic and something is wrong with the measurement system. The source of error should be located and the appropriate corrective action taken. Laboratories must monitor through QA channels the application of the sporadic marginal exceedance allowance to the LCS results to ensure random behavior.

TABLE 3.2
Precision and Accuracy Values
for MS/MSDs and LCS/LCSDs

| 8260B/5030 VOA Water Analyte | LCS and MS Lower Control Limit | LCS and MS Upper Control Limit | LCS and MS Lower ME Limit | LCS and MS Upper ME Limit | MSD %RPD |
|--|---------------------------------------|---------------------------------------|----------------------------------|----------------------------------|-----------------|
| 1,1,1,2-Tetrachloroethane | 80 | 130 | 75 | 135 | 0 - 30 |
| 1,1,1-Trichloroethane | 65 | 130 | 55 | 145 | 0 - 30 |
| 1,1,2,2-Tetrachloroethane | 65 | 130 | 55 | 140 | 0 - 30 |
| 1,1,2-Trichloroethane | 75 | 125 | 65 | 135 | 0 - 30 |
| 1,1-Dichloroethane | 70 | 135 | 60 | 145 | 0 - 30 |
| 1,1-Dichloroethylene* | 70 | 130 | 55 | 140 | 0 - 30 |
| 1,1-Dichloropropene | 75 | 130 | 65 | 140 | 0 - 30 |
| 1,2,3-Trichlorobenzene | 55 | 140 | 45 | 155 | 0 - 30 |
| 1,2,3-Trichloropropane | 75 | 125 | 65 | 130 | 0 - 30 |
| 1,2,4-Trichlorobenzene | 65 | 135 | 55 | 145 | 0 - 30 |
| 1,2,4-Trimethylbenzene | 75 | 130 | 65 | 140 | 0 - 30 |
| 1,2-Dibromo-3-chloropropane | 50 | 130 | 35 | 145 | 0 - 30 |
| 1,2-Dibromoethane (Ethylene Dibromide) | 80 | 120 | 75 | 125 | 0 - 30 |
| 1,2-Dichlorobenzene | 70 | 120 | 60 | 130 | 0 - 30 |
| 1,2-Dichloroethane* | 70 | 130 | 60 | 140 | 0 - 30 |
| 1,2-Dichloropropane | 75 | 125 | 65 | 135 | 0 - 30 |
| 1,3,5-Trimethylbenzene | 75 | 130 | 65 | 140 | 0 - 30 |
| 1,3-Dichlorobenzene | 75 | 125 | 65 | 130 | 0 - 30 |
| 1,3-Dichloropropane | 75 | 125 | 65 | 135 | 0 - 30 |
| 1,4-Dichlorobenzene | 75 | 125 | 65 | 130 | 0 - 30 |
| 2,2-Dichloropropane | 70 | 135 | 60 | 150 | 0 - 30 |
| 2-Butanone (MEK)* | 30 | 150 | 10 | 170 | 0 - 30 |
| 2-Chlorotoluene | 75 | 125 | 65 | 135 | 0 - 30 |
| 2-Hexanone | 55 | 130 | 45 | 140 | 0 - 30 |
| 4-Chlorotoluene | 75 | 130 | 65 | 135 | 0 - 30 |
| 4-Methyl-2-pentanone (MIBK) | 60 | 135 | 45 | 145 | 0 - 30 |
| Acetone | 40 | 140 | 20 | 160 | 0 - 30 |
| Benzene* | 80 | 120 | 75 | 130 | 0 - 30 |
| Bromobenzene | 75 | 125 | 70 | 130 | 0 - 30 |
| Bromochloromethane | 65 | 130 | 55 | 140 | 0 - 30 |
| Bromodichloromethane | 75 | 120 | 70 | 130 | 0 - 30 |
| Bromoform | 70 | 130 | 60 | 140 | 0 - 30 |
| Bromomethane (Methyl Bromide) | 30 | 145 | 10 | 165 | 0 - 30 |
| Carbon disulfide | 35 | 160 | 15 | 185 | 0 - 30 |
| Carbon tetrachloride* | 65 | 140 | 55 | 150 | 0 - 30 |
| Chlorobenzene | 80 | 120 | 75 | 130 | 0 - 30 |
| Chlorodibromomethane | 60 | 135 | 45 | 145 | 0 - 30 |
| Chloroethane | 60 | 135 | 50 | 145 | 0 - 30 |
| Chloroform* | 65 | 135 | 50 | 150 | 0 - 30 |
| Chloromethane (Methyl Chloride) | 40 | 125 | 25 | 140 | 0 - 30 |
| cis-1,2-Dichloroethylene | 70 | 125 | 60 | 135 | 0 - 30 |
| cis-1,3-Dichloropropylene | 70 | 130 | 60 | 140 | 0 - 30 |
| Dibromomethane | 75 | 125 | 65 | 135 | 0 - 30 |
| Dichlorodifluoromethane | 30 | 155 | 10 | 175 | 0 - 30 |
| Ethylbenzene | 75 | 125 | 65 | 135 | 0 - 30 |
| Hexachlorobutadiene | 50 | 140 | 35 | 160 | 0 - 30 |
| Isopropylbenzene | 75 | 125 | 65 | 135 | 0 - 30 |
| m,p-Xylene | 75 | 130 | 65 | 135 | 0 - 30 |
| Methyl Tert-butyl Ether (MTBE) | 65 | 125 | 55 | 135 | 0 - 30 |
| Methylene chloride | 55 | 140 | 40 | 155 | 0 - 30 |
| Naphthalene | 55 | 140 | 40 | 150 | 0 - 30 |
| n-Butylbenzene | 70 | 135 | 55 | 150 | 0 - 30 |
| n-Propylbenzene | 70 | 130 | 65 | 140 | 0 - 30 |
| o-Xylene | 80 | 120 | 75 | 130 | 0 - 30 |
| p-Isopropyltoluene | 75 | 130 | 65 | 140 | 0 - 30 |
| sec-Butylbenzene | 70 | 125 | 65 | 135 | 0 - 30 |
| Styrene | 65 | 135 | 65 | 145 | 0 - 30 |
| tert-Butylbenzene | 70 | 130 | 60 | 140 | 0 - 30 |
| Tetrachloroethene* | 45 | 150 | 25 | 165 | 0 - 30 |
| Toluene* | 75 | 120 | 70 | 130 | 0 - 30 |
| trans-1,2-Dichloroethene | 60 | 140 | 45 | 150 | 0 - 30 |
| trans-1,3-Dichloropropene | 55 | 140 | 40 | 155 | 0 - 30 |
| Trichloroethylene* | 70 | 125 | 60 | 135 | 0 - 30 |
| Trichlorofluoromethane | 60 | 145 | 45 | 160 | 0 - 30 |
| Vinyl Acetate | 66.8 | 136 | 50 | 150 | 0 - 30 |
| Vinyl chloride* | 50 | 145 | 35 | 165 | 0 - 30 |
| Xylene (Total) | 70 | 130 | 65 | 135 | 0 - 30 |

* TCLP Target Analyte

TABLE 3.2
Precision and Accuracy Values
for MS/MSDs and LCS/LCSDs

| | LCS | LCS | MS | MS | MSD |
|---------------------------|----------------------------|----------------------------|----------------------------|----------------------------|-------------|
| Metals 6010B Water | Lower Control Limit | Upper Control Limit | Lower Control Limit | Upper Control Limit | %RPD |
| Analyte | | | | | |
| Aluminum | 80 | 120 | 75 | 125 | 0 - 20 |
| Antimony | 80 | 120 | 75 | 125 | 0 - 20 |
| Arsenic | 80 | 120 | 75 | 125 | 0 - 20 |
| Barium | 80 | 120 | 75 | 125 | 0 - 20 |
| Beryllium | 80 | 120 | 75 | 125 | 0 - 20 |
| Cadmium | 80 | 120 | 75 | 125 | 0 - 20 |
| Calcium | 80 | 120 | 75 | 125 | 0 - 20 |
| Chromium | 80 | 120 | 75 | 125 | 0 - 20 |
| Cobalt | 80 | 120 | 75 | 125 | 0 - 20 |
| Copper | 80 | 120 | 75 | 125 | 0 - 20 |
| Iron | 80 | 120 | 75 | 125 | 0 - 20 |
| Lead | 80 | 120 | 75 | 125 | 0 - 20 |
| Magnesium | 80 | 120 | 75 | 125 | 0 - 20 |
| Manganese | 80 | 120 | 75 | 125 | 0 - 20 |
| Molybdenum | 80 | 120 | 75 | 125 | 0 - 20 |
| Nickel | 80 | 120 | 75 | 125 | 0 - 20 |
| Potassium | 80 | 120 | 75 | 125 | 0 - 20 |
| Selenium | 80 | 120 | 75 | 125 | 0 - 20 |
| Silver | 80 | 120 | 75 | 125 | 0 - 20 |
| Sodium | 80 | 120 | 75 | 125 | 0 - 20 |
| Thallium | 80 | 120 | 75 | 125 | 0 - 20 |
| Vanadium | 80 | 120 | 75 | 125 | 0 - 20 |
| Zinc | 80 | 120 | 75 | 125 | 0 - 20 |
| Metals 7470A Water | | | | | |
| Mercury | 80 | 120 | 75 | 125 | 0 - 20 |
| Metals 7471A Soil | | | | | |
| Mercury | 80 | 120 | 75 | 125 | 0 - 20 |
| Metals 6010B Soil | LCS | LCS | MS | MS | MSD |
| | Lower Control Limit | Upper Control Limit | Lower Control Limit | Upper Control Limit | %RPD |
| Analyte | | | | | |
| Aluminum | 80 | 120 | 75 | 125 | 0 - 20 |
| Antimony | 80 | 120 | 75 | 125 | 0 - 20 |
| Arsenic | 80 | 120 | 75 | 125 | 0 - 20 |
| Barium | 80 | 120 | 75 | 125 | 0 - 20 |
| Beryllium | 80 | 120 | 75 | 125 | 0 - 20 |
| Cadmium | 80 | 120 | 75 | 125 | 0 - 20 |
| Calcium | 80 | 120 | 75 | 125 | 0 - 20 |
| Chromium | 80 | 120 | 75 | 125 | 0 - 20 |
| Cobalt | 80 | 120 | 75 | 125 | 0 - 20 |
| Copper | 80 | 120 | 75 | 125 | 0 - 20 |
| Iron | 80 | 120 | 75 | 125 | 0 - 20 |
| Lead | 80 | 120 | 75 | 125 | 0 - 20 |
| Magnesium | 80 | 120 | 75 | 125 | 0 - 20 |
| Manganese | 80 | 120 | 75 | 125 | 0 - 20 |
| Molybdenum | 80 | 120 | 75 | 125 | 0 - 20 |
| Nickel | 80 | 120 | 75 | 125 | 0 - 20 |
| Potassium | 80 | 120 | 75 | 125 | 0 - 20 |
| Selenium | 80 | 120 | 75 | 125 | 0 - 20 |
| Silver | 75 | 120 | 75 | 125 | 0 - 20 |
| Sodium | 80 | 120 | 75 | 125 | 0 - 20 |
| Thallium | 80 | 120 | 75 | 125 | 0 - 20 |
| Vanadium | 80 | 120 | 75 | 125 | 0 - 20 |
| Zinc | 80 | 120 | 75 | 125 | 0 - 20 |

Table 3.2
Precision and Accuracy Values
for MS/MSDs and LCS/LCSDs

D.2 Marginal Exceedance

DoD will allow a number of sporadic marginal exceedances of the LCS-CLs. The number of exceedances is based on the total number of analytes spiked in the LCS. As the number of analytes in the LCS increases, more marginal exceedances are allowed. The number of allowable marginal exceedances is based on a policy decision that no more than 5% of the total number of analytes spiked in the LCS may exceed the DoD limits. Table D-1 presents the allowable number of marginal exceedances for a given number of analytes in the LCS.

TABLE D-1. NUMBER OF MARGINAL EXCEEDANCES

| Number of Analytes in LCS | Number of Marginal Exceedances of LCS-CLs |
|---------------------------|---|
| >90 | 5 |
| 71–90 | 4 |
| 51–70 | 3 |
| 31–50 | 2 |
| 11–30 | 1 |
| <11 | 0 |

A marginal exceedance is defined as beyond the LCS-CL but still within the marginal exceedance limits (set at 4 standard deviations around the mean). This outside boundary prevents a grossly out-of-control LCS from passing.

DoD requires that the marginal exceedances be sporadic (i.e., random). If the same analyte exceeds the LCS-CL repeatedly (e.g., 2 out of 3 consecutive LCS), that is an indication that the problem is systemic and something is wrong with the measurement system. The source of error should be located and the appropriate corrective action taken. Laboratories must monitor through QA channels the application of the sporadic marginal exceedance allowance to the LCS results to ensure random behavior.

TABLE 3.2
Precision and Accuracy Values
for MS/MSDs and LCS/LCSDs

| Surrogate Recoveries | | |
|-----------------------------|----------------------------|----------------------------|
| Analyte | Lower Control Limit | Upper Control Limit |
| 8260B Water | | |
| 4-Bromofluorobenzene | 75 | 120 |
| Dibromofluoromethane | 85 | 115 |
| Toluene-d8 | 85 | 120 |
| 8260B Solid: | | |
| 4-Bromofluorobenzene | 85 | 120 |
| Dibromofluoromethane | 73 | 147 |
| Toluene-d8 | 85 | 115 |
| 8270C Water: | | |
| 2-Fluorobiphenyl | 50 | 110 |
| Terphenyl-d14 | 50 | 135 |
| 2,4,6-Tribromophenol | 40 | 125 |
| 2-Fluorophenol | 20 | 110 |
| Nitrobenzene-d5 | 40 | 110 |
| 8270C Solid: | | |
| 2-Fluorobiphenyl | 45 | 105 |
| Terphenyl-d14 | 30 | 125 |
| 2,4,6-Tribromophenol | 35 | 125 |
| 2-Fluorophenol | 35 | 105 |
| Phenol-d5/d6 | 40 | 100 |
| Nitrobenzene-d5 | 35 | 100 |
| 8081A Water: | | |
| Decachlorobiphenyl | 30 | 135 |
| TCMX | 25 | 140 |
| 8081A Solid: | | |
| Decachlorobiphenyl | 55 | 130 |
| TCMX | 70 | 125 |
| 8082 Water: | | |
| Decachlorobiphenyl | 40 | 135 |
| 8082 Solid: | | |
| Decachlorobiphenyl | 60 | 125 |

TABLE 3.2
Precision and Accuracy Values
for MS/MSDs and LCS/LCSDs

| BNA 8270C Soil | LCS and MS | LCS and MS | LCS and MS | LCS and MS | MSD |
|--|----------------------------|----------------------------|-----------------------|-----------------------|-------------|
| Analyte | Lower Control Limit | Upper Control Limit | Lower ME Limit | Upper ME Limit | %RPD |
| 1, 1'-Biphenyl | 50 | 150 | 35 | 150 | 0 - 30 |
| 1,2,4-Trichlorobenzene | 45 | 110 | 30 | 120 | 0 - 30 |
| 1,2-Dichlorobenzene | 45 | 95 | 35 | 135 | 0 - 30 |
| 1,2-Diphenylhydrazine | 10 | 150 | 0 | 160 | 0 - 30 |
| 1,3-Dichlorobenzene | 40 | 100 | 30 | 110 | 0 - 30 |
| 1,4-Dichlorobenzene | 35 | 105 | 25 | 115 | 0 - 30 |
| 2,4,5-Trichlorophenol* | 50 | 110 | 40 | 120 | 0 - 30 |
| 2,4,6-Trichlorophenol* | 45 | 110 | 30 | 120 | 0 - 30 |
| 2,4-Dichlorophenol | 45 | 110 | 35 | 120 | 0 - 30 |
| 2,4-Dimethylphenol | 30 | 105 | 20 | 115 | 0 - 30 |
| 2,4-Dinitrophenol | 15 | 130 | 10 | 150 | 0 - 30 |
| 2,4-Dinitrotoluene* | 50 | 115 | 35 | 130 | 0 - 30 |
| 2,6-Dichlorophenol | 10 | 150 | 0 | 160 | 0 - 30 |
| 2,6-Dinitrotoluene | 50 | 110 | 35 | 125 | 0 - 30 |
| 2-Chloronaphthalene | 45 | 105 | 35 | 115 | 0 - 30 |
| 2-Chlorophenol | 45 | 105 | 35 | 115 | 0 - 30 |
| 2-Methyl-4,6-dinitrophenol | 30 | 135 | 10 | 155 | 0 - 30 |
| 2-Methylnaphthalene | 45 | 105 | 35 | 115 | 0 - 30 |
| 2-Methylphenol (o-Cresol)* | 40 | 105 | 30 | 115 | 0 - 30 |
| 2-Nitroaniline (o-Nitroaniline) | 45 | 120 | 30 | 130 | 0 - 30 |
| 2-Nitrophenol | 40 | 110 | 30 | 120 | 0 - 30 |
| 3,3'Dichlorobenzidine | 10 | 130 | 0 | 145 | 0 - 30 |
| 3-Methylphenol/4-Methylphenol (m,p-Cresol) | 40 | 105 | 30 | 120 | 0 - 30 |
| 3-Nitroaniline (m-Nitroaniline) | 25 | 110 | 15 | 125 | 0 - 30 |
| 4-Bromophenyl phenyl ether | 45 | 115 | 35 | 130 | 0 - 30 |
| 4-Chloro-3-methylphenol | 45 | 115 | 35 | 125 | 0 - 30 |
| 4-Chloroaniline | 10 | 95 | 0 | 110 | 0 - 30 |
| 4-Chlorophenyl phenyl ether | 45 | 110 | 35 | 120 | 0 - 30 |
| 4-Nitroaniline (p-Nitroaniline) | 35 | 115 | 20 | 125 | 0 - 30 |
| 4-Nitrophenol | 15 | 140 | 10 | 160 | 0 - 30 |
| Acenaphthene | 45 | 110 | 35 | 120 | 0 - 30 |
| Acenaphthylene | 45 | 105 | 35 | 115 | 0 - 30 |
| Acetophenone | 27 | 95 | 10 | 115 | 0 - 30 |
| Anthracene | 55 | 105 | 45 | 115 | 0 - 30 |
| Atrazine | 10 | 150 | 0 | 160 | 0 - 30 |
| Benz(a)anthracene | 50 | 110 | 40 | 120 | 0 - 30 |
| Benzaldehyde | 50 | 150 | 35 | 150 | 0 - 30 |
| Benzidine | 10 | 150 | 0 | 160 | 0 - 30 |
| Benzo(a)pyrene | 50 | 110 | 40 | 120 | 0 - 30 |
| Benzo(b)fluoranthene | 45 | 115 | 35 | 125 | 0 - 30 |
| Benzo(g,h,i)perylene | 40 | 125 | 25 | 140 | 0 - 30 |
| Benzo(k)fluoranthene | 45 | 125 | 30 | 135 | 0 - 30 |
| Benzoic Acid | 0 | 110 | 0 | 130 | 0 - 30 |
| Benzyl alcohol | 20 | 125 | 10 | 140 | 0 - 30 |
| Bis(2-chlorethoxy)methane | 45 | 110 | 30 | 120 | 0 - 30 |
| Bis(2-chloroethyl) ether | 40 | 105 | 25 | 115 | 0 - 30 |
| Bis(2-chloroisopropyl) ether | 20 | 115 | 10 | 130 | 0 - 30 |
| Bis(2-ethylhexyl) phthalate | 45 | 125 | 35 | 140 | 0 - 30 |
| Butyl benzyl phthalate | 50 | 125 | 35 | 135 | 0 - 30 |
| Caprolactam | 50 | 150 | 35 | 150 | 0 - 30 |
| Carbazole | 45 | 115 | 30 | 130 | 0 - 30 |
| Chrysene | 55 | 110 | 45 | 120 | 0 - 30 |
| Dibenz(a,h)anthracene | 40 | 125 | 25 | 140 | 0 - 30 |
| Dibenzofuran | 50 | 105 | 40 | 110 | 0 - 30 |
| Diethyl phthalate | 50 | 115 | 40 | 125 | 0 - 30 |
| Dimethyl phthalate | 50 | 110 | 40 | 120 | 0 - 30 |
| Di-n-butyl phthalate | 55 | 110 | 45 | 120 | 0 - 30 |
| Di-n-octyl phthalate | 40 | 130 | 25 | 145 | 0 - 30 |
| Diphenylamine | 50 | 115 | 40 | 125 | 0 - 30 |
| Fluoranthene | 55 | 115 | 45 | 125 | 0 - 30 |
| Fluorene | 50 | 110 | 40 | 115 | 0 - 30 |
| Hexachlorobenzene* | 45 | 120 | 35 | 130 | 0 - 30 |
| Hexachlorobutadiene* | 40 | 115 | 25 | 130 | 0 - 30 |
| Hexachlorocyclopentadiene | 18 | 110 | 5 | 120 | 0 - 30 |
| Hexachloroethane* | 35 | 110 | 20 | 120 | 0 - 30 |
| Indeno(1,2,3-cd)pyrene | 40 | 120 | 25 | 135 | 0 - 30 |
| Isophorone | 45 | 110 | 30 | 125 | 0 - 30 |
| Naphthalene | 40 | 105 | 30 | 120 | 0 - 30 |
| Nitrobenzene* | 40 | 115 | 30 | 125 | 0 - 30 |
| N-Nitrosodimethylamine | 20 | 115 | 10 | 130 | 0 - 30 |
| N-Nitrosodi-n-propylamine | 40 | 115 | 30 | 125 | 0 - 30 |
| N-Nitrosodiphenylamine | 50 | 115 | 40 | 125 | 0 - 30 |
| N-Nitrosopyrrolidine | 10 | 150 | 0 | 106 | 0 - 30 |
| Pentachlorophenol* | 25 | 120 | 10 | 135 | 0 - 30 |
| Phenanthrene | 50 | 110 | 40 | 120 | 0 - 30 |
| Phenol | 40 | 100 | 30 | 110 | 0 - 30 |
| Pyrene | 45 | 125 | 35 | 135 | 0 - 30 |

TABLE 3.2
Precision and Accuracy Values
for MS/MSDs and LCS/LCSDs

| BNA 8270C Water | LCS and MS | LCS and MS | LCS and MS | LCS and MS | MSD |
|--|----------------------------|----------------------------|-----------------------|-----------------------|-------------|
| Analyte | Lower Control Limit | Upper Control Limit | Lower ME Limit | Upper ME Limit | %RPD |
| 1, 1'-Biphenyl | 50 | 150 | 35 | 150 | 0 - 30 |
| 1,2,4-Trichlorobenzene | 53 | 96 | 40 | 120 | 0 - 30 |
| 1,2-Dichlorobenzene | 35 | 100 | 20 | 115 | 0 - 30 |
| 1,2-Diphenylhydrazine | 55 | 115 | 45 | 120 | 0 - 30 |
| 1,3-Dichlorobenzene | 30 | 100 | 20 | 110 | 0 - 30 |
| 1,4-Dichlorobenzene | 30 | 100 | 20 | 110 | 0 - 30 |
| 2,4,5-Trichlorophenol* | 50 | 110 | 40 | 120 | 0 - 30 |
| 2,4,6-Trichlorophenol* | 50 | 115 | 40 | 125 | 0 - 30 |
| 2,4-Dichlorophenol | 50 | 105 | 40 | 115 | 0 - 30 |
| 2,4-Dimethylphenol | 30 | 110 | 15 | 125 | 0 - 30 |
| 2,4-Dinitrophenol | 15 | 140 | 10 | 160 | 0 - 30 |
| 2,4-Dinitrotoluene* | 50 | 120 | 40 | 130 | 0 - 30 |
| 2,6-Dichlorophenol | 10 | 150 | 0 | 160 | 0 - 30 |
| 2,6-Dinitrotoluene | 50 | 115 | 35 | 130 | 0 - 30 |
| 2-Chloronaphthalene | 50 | 105 | 40 | 115 | 0 - 30 |
| 2-Chlorophenol | 35 | 105 | 25 | 115 | 0 - 30 |
| 2-Methyl-4,6-dinitrophenol | 40 | 130 | 25 | 145 | 0 - 30 |
| 2-Methylnaphthalene | 45 | 105 | 35 | 115 | 0 - 30 |
| 2-Methylphenol (o-Cresol)* | 40 | 110 | 25 | 120 | 0 - 30 |
| 2-Nitroaniline (o-Nitroaniline) | 50 | 115 | 35 | 125 | 0 - 30 |
| 2-Nitrophenol | 40 | 115 | 25 | 125 | 0 - 30 |
| 3,3'-Dichlorobenzidine | 20 | 110 | 10 | 125 | 0 - 30 |
| 3-Methylphenol/4-Methylphenol (m,p-Cresol) | 30 | 110 | 20 | 125 | 0 - 30 |
| 3-Nitroaniline (m-Nitroaniline) | 20 | 125 | 10 | 145 | 0 - 30 |
| 4-Bromophenyl phenyl ether | 50 | 115 | 40 | 125 | 0 - 30 |
| 4-Chloro-3-methylphenol | 45 | 110 | 35 | 120 | 0 - 30 |
| 4-Chloroaniline | 15 | 110 | 10 | 125 | 0 - 30 |
| 4-Chlorophenyl phenyl ether | 50 | 110 | 40 | 120 | 0 - 30 |
| 4-Nitroaniline (p-Nitroaniline) | 35 | 120 | 20 | 130 | 0 - 30 |
| 4-Nitrophenol | 0 | 125 | 0 | 145 | 0 - 30 |
| Acenaphthene | 45 | 110 | 35 | 120 | 0 - 30 |
| Acenaphthylene | 50 | 105 | 40 | 115 | 0 - 30 |
| Acetophenone | 57 | 108 | 45 | 120 | 0 - 30 |
| Anthracene | 55 | 110 | 45 | 120 | 0 - 30 |
| Atrazine | 10 | 150 | 0 | 160 | 0 - 30 |
| Benz(a)anthracene | 55 | 110 | 45 | 120 | 0 - 30 |
| Benzaldehyde | 50 | 150 | 35 | 150 | 0 - 30 |
| Benzidine | 10 | 150 | | | 0 - 30 |
| Benzo(a)pyrene | 55 | 110 | 45 | 120 | 0 - 30 |
| Benzo(b)fluoranthene | 45 | 120 | 35 | 130 | 0 - 30 |
| Benzo(g,h,i)perylene | 40 | 125 | 25 | 135 | 0 - 30 |
| Benzo(k)fluoranthene | 45 | 125 | 30 | 135 | 0 - 30 |
| Benzoic Acid | 0 | 125 | 0 | 150 | 0 - 30 |
| Benzyl alcohol | 30 | 110 | 15 | 125 | 0 - 30 |
| Bis(2-chloroethoxy)methane | 45 | 105 | 35 | 115 | 0 - 30 |
| Bis(2-chloroethyl) ether | 35 | 110 | 25 | 120 | 0 - 30 |
| Bis(2-chloroisopropyl) ether | 25 | 130 | 10 | 150 | 0 - 30 |
| Bis(2-ethylhexyl) phthalate | 40 | 125 | 30 | 140 | 0 - 30 |
| Butyl benzyl phthalate | 45 | 115 | 35 | 130 | 0 - 30 |
| Caprolactam | 50 | 150 | 35 | 150 | 0 - 30 |
| Carbazole | 50 | 115 | 35 | 130 | 0 - 30 |
| Chrysene | 55 | 110 | 45 | 120 | 0 - 30 |
| Dibenz(a,h)anthracene | 40 | 125 | 30 | 140 | 0 - 30 |
| Dibenzofuran | 55 | 105 | 45 | 115 | 0 - 30 |
| Diethyl phthalate | 40 | 120 | 30 | 130 | 0 - 30 |
| Dimethyl phthalate | 25 | 125 | 10 | 145 | 0 - 30 |
| Di-n-butyl phthalate | 55 | 115 | 45 | 125 | 0 - 30 |
| Di-n-octyl phthalate | 35 | 135 | 20 | 155 | 0 - 30 |
| Diphenylamine | 50 | 110 | 35 | 120 | 0 - 30 |
| Fluoranthene | 55 | 115 | 45 | 125 | 0 - 30 |
| Fluorene | 50 | 110 | 40 | 120 | 0 - 30 |
| Hexachlorobenzene* | 50 | 110 | 40 | 120 | 0 - 30 |
| Hexachlorobutadiene* | 25 | 105 | 15 | 115 | 0 - 30 |
| Hexachlorocyclopentadiene | 19 | 107 | 10 | 120 | 0 - 30 |
| Hexachloroethane* | 30 | 95 | 15 | 105 | 0 - 30 |
| Indeno(1,2,3-cd)pyrene | 45 | 125 | 30 | 140 | 0 - 30 |
| Isophorone | 50 | 110 | 40 | 125 | 0 - 30 |
| Naphthalene | 40 | 100 | 30 | 115 | 0 - 30 |
| Nitrobenzene* | 45 | 110 | 35 | 120 | 0 - 30 |
| N-Nitrosodimethylamine | 25 | 110 | 10 | 125 | 0 - 30 |
| N-Nitrosodi-n-propylamine | 35 | 130 | 20 | 145 | 0 - 30 |
| N-Nitrosodiphenylamine | 50 | 110 | 35 | 120 | 0 - 30 |
| N-Nitrosopyrrolidine | 10 | 150 | 0 | 160 | 0 - 30 |
| Pentachlorophenol* | 40 | 115 | 25 | 130 | 0 - 30 |
| Phenanthrene | 50 | 115 | 40 | 130 | 0 - 30 |
| Phenol | 0 | 115 | 0 | 135 | 0 - 30 |
| Pyrene | 50 | 130 | 35 | 140 | 0 - 30 |
| | | | | | 0 - 30 |
| | | | | | 0 - 30 |

* TCLP Target Analyte

Table 3.2
Precision and Accuracy Values
for MS/MSDs and LCS/LCSDs

* TCLP Target Analyte

D.2 Marginal Exceedance

DoD will allow a number of sporadic marginal exceedances of the LCS-CLs. The number of exceedances is based on the total number of analytes spiked in the LCS. As the number of analytes in the LCS increases, more marginal exceedances are allowed. The number of allowable marginal exceedances is based on a policy decision that no more than 5% of the total number of analytes spiked in the LCS may exceed the DoD limits. Table D-1 presents the allowable number of marginal exceedances for a given number of analytes in the LCS.

TABLE D-1. NUMBER OF MARGINAL EXCEEDANCES

Number of Analytes in LCS

>90
 71–90
 51–70
 31–50
 11–30
 <11

A marginal exceedance is defined as beyond the LCS-CL but still within the marginal exceedance limits (set at 4 standard deviations around the mean). This outside boundary prevents a grossly out-of-control LCS from passing.

DoD requires that the marginal exceedances be sporadic (i.e., random). If the same analyte exceeds the LCS-CL repeatedly (e.g., 2 out of 3 consecutive LCS), that is an indication that the problem is systemic and something is wrong with the measurement system. The source of error should be located and the appropriate corrective action taken.

TABLE 3.2
Precision and Accuracy Values
for MS/MSDs and LCS/LCSDs

| | LCS | | MS | | MSD |
|---------------------------|----------------------------|----------------------------|----------------------------|----------------------------|-------------|
| Metals 6010B Water | Lower Control Limit | Upper Control Limit | Lower Control Limit | Upper Control Limit | %RPD |
| Analyte | | | | | |
| Aluminum | 80 | 120 | 75 | 125 | 0 - 20 |
| Antimony | 80 | 120 | 75 | 125 | 0 - 20 |
| Arsenic | 80 | 120 | 75 | 125 | 0 - 20 |
| Barium | 80 | 120 | 75 | 125 | 0 - 20 |
| Beryllium | 80 | 120 | 75 | 125 | 0 - 20 |
| Cadmium | 80 | 120 | 75 | 125 | 0 - 20 |
| Calcium | 80 | 120 | 75 | 125 | 0 - 20 |
| Chromium | 80 | 120 | 75 | 125 | 0 - 20 |
| Cobalt | 80 | 120 | 75 | 125 | 0 - 20 |
| Copper | 80 | 120 | 75 | 125 | 0 - 20 |
| Iron | 80 | 120 | 75 | 125 | 0 - 20 |
| Lead | 80 | 120 | 75 | 125 | 0 - 20 |
| Magnesium | 80 | 120 | 75 | 125 | 0 - 20 |
| Manganese | 80 | 120 | 75 | 125 | 0 - 20 |
| Molybdenum | 80 | 120 | 75 | 125 | 0 - 20 |
| Nickel | 80 | 120 | 75 | 125 | 0 - 20 |
| Potassium | 80 | 120 | 75 | 125 | 0 - 20 |
| Selenium | 80 | 120 | 75 | 125 | 0 - 20 |
| Silver | 80 | 120 | 75 | 125 | 0 - 20 |
| Sodium | 80 | 120 | 75 | 125 | 0 - 20 |
| Thallium | 80 | 120 | 75 | 125 | 0 - 20 |
| Vanadium | 80 | 120 | 75 | 125 | 0 - 20 |
| Zinc | 80 | 120 | 75 | 125 | 0 - 20 |
| Metals 7470A Water | | | | | |
| Mercury | 80 | 120 | 75 | 125 | 0 - 20 |
| Metals 7471A Soil | | | | | |
| Mercury | 80 | 120 | 75 | 125 | 0 - 20 |
| Metals 6010B Soil | LCS | LCS | MS | MS | MSD |
| Analyte | Lower Control Limit | Upper Control Limit | Lower Control Limit | Upper Control Limit | %RPD |
| Aluminum | 80 | 120 | 75 | 125 | 0 - 20 |
| Antimony | 80 | 120 | 75 | 125 | 0 - 20 |
| Arsenic | 80 | 120 | 75 | 125 | 0 - 20 |
| Barium | 80 | 120 | 75 | 125 | 0 - 20 |
| Beryllium | 80 | 120 | 75 | 125 | 0 - 20 |
| Cadmium | 80 | 120 | 75 | 125 | 0 - 20 |
| Calcium | 80 | 120 | 75 | 125 | 0 - 20 |
| Chromium | 80 | 120 | 75 | 125 | 0 - 20 |
| Cobalt | 80 | 120 | 75 | 125 | 0 - 20 |
| Copper | 80 | 120 | 75 | 125 | 0 - 20 |
| Iron | 80 | 120 | 75 | 125 | 0 - 20 |
| Lead | 80 | 120 | 75 | 125 | 0 - 20 |
| Magnesium | 80 | 120 | 75 | 125 | 0 - 20 |
| Manganese | 80 | 120 | 75 | 125 | 0 - 20 |
| Molybdenum | 80 | 120 | 75 | 125 | 0 - 20 |
| Nickel | 80 | 120 | 75 | 125 | 0 - 20 |
| Potassium | 80 | 120 | 75 | 125 | 0 - 20 |
| Selenium | 80 | 120 | 75 | 125 | 0 - 20 |
| Silver | 75 | 120 | 75 | 125 | 0 - 20 |
| Sodium | 80 | 120 | 75 | 125 | 0 - 20 |
| Thallium | 80 | 120 | 75 | 125 | 0 - 20 |
| Vanadium | 80 | 120 | 75 | 125 | 0 - 20 |
| Zinc | 80 | 120 | 75 | 125 | 0 - 20 |

TABLE 3.2
Precision and Accuracy Values
for MS/MSDs and LCS/LCSDs

| PCB 8082 Water | LCS and MS | LCS and MS | MSD |
|--------------------------|----------------------------|----------------------------|----------------|
| Analyte | Lower Control Limit | Upper Control Limit | %RPD |
| Aroclor 1016 | 25 | 145 | 0 - 30 |
| Aroclor 1260 | 30 | 145 | 0 - 30 |
| PCB 8082 Soil | LCS and MS | LCS and MS | MSD |
| Analyte | Lower Control Limit | Upper Control Limit | %RPD |
| Aroclor 1016 | 40 | 140 | 0 - 30 |
| Aroclor 1260 | 60 | 130 | 0 - 30 |

TABLE 3.2
Precision and Accuracy Values
for MS/MSDs and LCS/LCSDs

| 8081A Water | LCS and MS | LCS and MS | LCS and MS | LCS and MS | MSD |
|---------------------|----------------------------|----------------------------|-----------------------|-----------------------|-------------|
| Analyte | Lower Control Limit | Upper Control Limit | Lower ME Limit | Upper ME Limit | %RPD |
| 4,4'-DDD | 25 | 150 | 10 | 170 | 0 - 30 |
| 4,4'-DDE | 35 | 140 | 15 | 160 | 0 - 30 |
| 4,4'-DDT | 45 | 140 | 30 | 155 | 0 - 30 |
| Aldrin | 25 | 140 | 10 | 155 | 0 - 30 |
| alpha-BHC | 60 | 130 | 50 | 140 | 0 - 30 |
| alpha-Chlordane | 65 | 125 | 55 | 135 | 0 - 30 |
| beta-BHC | 65 | 125 | 55 | 135 | 0 - 30 |
| delta-BHC | 45 | 135 | 30 | 150 | 0 - 30 |
| Dieldrin | 60 | 130 | 50 | 140 | 0 - 30 |
| Endosulfan I | 50 | 110 | 40 | 120 | 0 - 30 |
| Endosulfan II | 30 | 130 | 10 | 150 | 0 - 30 |
| Endosulfan sulfate | 55 | 135 | 40 | 150 | 0 - 30 |
| Endrin* | 55 | 135 | 45 | 145 | 0 - 30 |
| Endrin aldehyde | 55 | 135 | 40 | 150 | 0 - 30 |
| Endrin ketone | 75 | 125 | 70 | 135 | 0 - 30 |
| gamma-BHC* | 25 | 135 | 10 | 155 | 0 - 30 |
| gamma-Chlordane | 60 | 125 | 50 | 135 | 0 - 30 |
| Heptachlor* | 40 | 130 | 30 | 145 | 0 - 30 |
| Heptachlor epoxide* | 60 | 130 | 50 | 140 | 0 - 30 |
| Methoxychlor* | 55 | 150 | 40 | 165 | 0 - 30 |
| Toxaphene* | 40 | 130 | 30 | 145 | 0 - 30 |

| Pest 8081A Soil | LCS and MS | LCS and MS | LCS and MS | LCS and MS | MSD |
|------------------------|----------------------------|----------------------------|-----------------------|-----------------------|-------------|
| Analyte | Lower Control Limit | Upper Control Limit | Lower ME Limit | Upper ME Limit | %RPD |
| 4,4'-DDD | 30 | 135 | 10 | 155 | 0 - 30 |
| 4,4'-DDE | 70 | 125 | 60 | 135 | 0 - 30 |
| 4,4'-DDT | 45 | 140 | 30 | 155 | 0 - 30 |
| Aldrin | 45 | 140 | 30 | 155 | 0 - 30 |
| alpha-BHC | 60 | 125 | 50 | 135 | 0 - 30 |
| alpha-Chlordane | 65 | 120 | 55 | 130 | 0 - 30 |
| Beta-BHC | 60 | 125 | 50 | 135 | 0 - 30 |
| delta-BHC | 55 | 130 | 45 | 145 | 0 - 30 |
| Dieldrin | 65 | 125 | 55 | 135 | 0 - 30 |
| Endosulfan | 15 | 135 | 10 | 155 | 0 - 30 |
| Endosulfan | 35 | 140 | 20 | 160 | 0 - 30 |
| Endosulfan sulfate | 60 | 135 | 50 | 145 | 0 - 30 |
| Endrin | 60 | 135 | 50 | 145 | 0 - 30 |
| Endrin aldehyde | 35 | 145 | 20 | 165 | 0 - 30 |
| Endrin ketone | 65 | 135 | 55 | 145 | 0 - 30 |
| gamma-BHC | 60 | 125 | 50 | 135 | 0 - 30 |
| gamma-Chlordane | 65 | 125 | 55 | 135 | 0 - 30 |
| Heptachlor | 50 | 140 | 35 | 155 | 0 - 30 |
| Heptachlor epoxide | 65 | 130 | 55 | 140 | 0 - 30 |
| Methoxychlor | 55 | 145 | 45 | 155 | 0 - 30 |
| Toxaphene* | 40 | 130 | 30 | 145 | 0 - 30 |

* TCLP target analyte

Table 3.2
Precision and Accuracy Values
for MS/MSDs and LCS/LCSDs

D.2 Marginal Exceedance

DoD will allow a number of sporadic marginal exceedances of the LCS-CLs. The number of exceedances is based on the total number of analytes spiked in the LCS. As the number of analytes in the LCS increases, more marginal exceedances are allowed. The number of allowable marginal exceedances is based on a policy decision that no more than 5% of the total number of analytes spiked in the LCS may exceed the DoD limits. Table D-1 presents the allowable number of marginal exceedances for a given number of analytes in the LCS.

TABLE D-1. NUMBER OF MARGINAL EXCEEDANCES

| Number of Analytes in the LCS | | Number of Marginal Exceedances of LCS-CLs |
|-------------------------------|--|---|
| >90 | | 5 |
| 71–90 | | 4 |
| 51–70 | | 3 |
| 31–50 | | 2 |
| 11–30 | | 1 |
| <11 | | 0 |

A marginal exceedance is defined as beyond the LCS-CL but still within the marginal exceedance limits (set at 4 standard deviations around the mean). This outside boundary prevents a grossly out-of-control LCS from passing.

DoD requires that the marginal exceedances be sporadic (i.e., random). If the same analyte exceeds the LCS-CL repeatedly (e.g., 2 out of 3 consecutive LCS), that is an indication that the problem is systemic and something is wrong with the measurement system. The source of error should be located and the appropriate corrective action taken. Laboratories must monitor through QA channels the application of the sporadic marginal exceedance allowance to the LCS results to ensure random behavior.

TABLE 3.2
Precision and Accuracy Values
for MS/MSDs and LCS/LCSDs

| TCLP Herbicides 8151A Water | LCS and MS | LCS and MS | MSD |
|------------------------------------|----------------------------|----------------------------|-------------|
| Analyte | Lower Control Limit | Upper Control Limit | %RPD |
| 2,4-D | 35 | 115 | 0 - 30 |
| 2,4,5-TP (Silvex) | 50 | 115 | 0 - 30 |
| 2,4,5-T | 35 | 110 | 0 - 30 |

| TCLP Herbicides 8151A Soil | LCS and MS | LCS and MS | MSD |
|-----------------------------------|----------------------------|----------------------------|-------------|
| Analyte | Lower Control Limit | Upper Control Limit | %RPD |
| 2,4-D | 35 | 145 | 0 - 30 |
| 2,4,5-TP (Silvex) | 45 | 125 | 0 - 30 |

TABLE 3.2
Precision and Accuracy Values
for MS/MSDs and LCS/LCSDs

Precision and Accuracy Data

| | LCS Lower Control Limit | LCS Upper Control Limit | MS Lower Control Limit | MS Upper Control Limit | MSD/DUP %RPD |
|--|--|--|---------------------------------------|---------------------------------------|-------------------------|
| EPA 150.1 Water Analyte | | | | | |
| pH | 95 | 105 | N/A | N/A | 0 - 10 |
| SW846 9045C Solid Analyte | | | | | |
| Corrosivity | 95 | 105 | N/A | N/A | 0 - 10 |
| EPA 160.2 Water Analyte | | | | | |
| Total Suspended Solids | 95 | 105 | N/A | N/A | 0 - 10 |
| EPA 160.1 Water Analyte | | | | | |
| Total Dissolved Solids | 95 | 105 | N/A | N/A | 0 - 10 |
| EPA 410.4 Water Analyte | | | | | |
| COD | 85 | 115 | 85 | 115 | 0 - 20 |
| EPA 351.2 Water Analyte | | | | | |
| Nitrogen, Total Kjeldahl | 90 | 110 | 80 | 120 | 0 - 20 |
| EPA 351.2 Modified Soil Analyte | | | | | |
| Nitrogen, Total Kjeldahl | 90 | 110 | 75 | 125 | 0 - 20 |
| EPA 350.1 Water Analyte | | | | | |
| Ammonia | 90 | 110 | 80 | 120 | 0 - 20 |
| EPA 350.1 Modified Soil Analyte | | | | | |
| Ammonia | 90 | 110 | 75 | 125 | 0 - 20 |
| EPA 351.2/350.1 Water Analyte | | | | | |
| Total Organic Nitrogen | NA | NA | NA | NA | 0 - 20 |
| EPA 351.2/350.1 Modified Soil Analyte | | | | | |
| Total Organic Nitrogen | NA | NA | NA | NA | 0 - 30 |
| EPA 413.1 Water Analyte | | | | | |
| Oil & Grease | 76 | 106 | 70 | 130 | 0 - 20 |
| EPA 1664 Water Analyte | | | | | |
| Oil & Grease | 79 | 114 | 79 | 114 | 0 - 18 |

TABLE 3.2
Precision and Accuracy Values
for MS/MSDs and LCS/LCSDs

| | LCS Lower Control Limit | LCS Upper Control Limit | MS Lower Control Limit | MS Upper Control Limit | MSD/DUP %RPD |
|--------------------------------------|--|--|---------------------------------------|---------------------------------------|-------------------------|
| EPA 300.0 Water | | | | | |
| Analyte | | | | | |
| Nitrate | 90 | 110 | 80 | 120 | 0 - 20 |
| Nitrite | 90 | 110 | 80 | 120 | 0 - 20 |
| SW846 9056 Soil | | | | | |
| Analyte | | | | | |
| Nitrate | 90 | 110 | 80 | 120 | 0 - 20 |
| Nitrite | 90 | 110 | 80 | 120 | 0 - 20 |
| EPA 405.1 Water | | | | | |
| Analyte | | | | | |
| BOD, 5 DAY | 84 | 115 | N/A | N/A | N/A |
| SW 846 7196A Water | | | | | |
| Analyte | | | | | |
| Chromium, Hexavalent | 85 | 115 | 85 | 115 | 0 - 15 |
| SW 846 7196A Soil | | | | | |
| Analyte | | | | | |
| Chromium, Hexavalent | 75 | 115 | 50 | 125 | 0 - 30 |
| SW846 9010B/9012A Water | | | | | |
| Analyte | | | | | |
| Cyanide | 90 | 110 | 85 | 119 | 0 - 20 |
| SW846 9010B/9012A Soil | | | | | |
| Analyte | | | | | |
| Cyanide | 90 | 110 | 45 | 135 | 0 - 30 |
| SW846 9060 / Lloyd Kahn Water | | | | | |
| Analyte | | | | | |
| Total Organic Carbon | 85 | 115 | 80 | 120 | 0-20 |
| SW846 9060 / Lloyd Kahn Soil | | | | | |
| Analyte | | | | | |
| Total Organic Carbon | 80 | 120 | 66 | 121 | 0-25 |
| SW846 8015M Water | | | | | |
| Analyte | | | | | |
| Total Petroleum Hydrocarbon | 47 | 145 | 43 | 140 | 0-29 |
| SW846 8015M Soil | | | | | |
| Analyte | | | | | |
| Total Petroleum Hydrocarbon | 50 | 149 | 30 | 143 | 0-27 |

TABLE 3.2
Precision and Accuracy Values
for MS/MSDs and LCS/LCSDs

| | LCS Lower Control Limit | LCS Upper Control Limit | MS Lower Control Limit | MS Upper Control Limit | MSD/DUP %RPD |
|------------------------------------|--|--|---------------------------------------|---------------------------------------|-------------------------|
| EPA 330.5 Water Analyte | | | | | |
| Total Residual Chlorine | 85 | 115 | NA | NA | 0-20 |
| EPA 120.1 Water Analyte | | | | | |
| Specific Conductance | 95 | 105 | NA | NA | 0-10 |
| EPA 180.1 Water Analyte | | | | | |
| Turbidity | 86 | 118 | NA | NA | 0-20 |
| EPA 170.1 Water Analyte | | | | | |
| Temperature | NA | NA | NA | NA | NA |

Table 3.3
Organic Surrogate Recoveries

| Analyte | Lower Control Limit | Upper Control Limit |
|----------------------|----------------------------|----------------------------|
| 8260B Water | | |
| 4-Bromofluorobenzene | 75 | 120 |
| Dibromofluoromethane | 85 | 115 |
| Toluene-d8 | 85 | 120 |
| 8260B Solid: | | |
| 4-Bromofluorobenzene | 85 | 120 |
| Dibromofluoromethane | 73 | 147 |
| Toluene-d8 | 85 | 115 |
| 8270C Water: | | |
| 2-Fluorobiphenyl | 50 | 110 |
| Terphenyl-d14 | 50 | 135 |
| 2,4,6-Tribromophenol | 40 | 125 |
| 2-Fluorophenol | 20 | 110 |
| Nitrobenzene-d5 | 40 | 110 |
| 8270C Solid: | | |
| 2-Fluorobiphenyl | 45 | 105 |
| Terphenyl-d14 | 30 | 125 |
| 2,4,6-Tribromophenol | 35 | 125 |
| 2-Fluorophenol | 35 | 105 |
| Phenol-d5/d6 | 40 | 100 |
| Nitrobenzene-d5 | 35 | 100 |
| 8081A Water: | | |
| Decachlorobiphenyl | 30 | 135 |
| TCMX | 25 | 140 |
| 8081A Solid: | | |
| Decachlorobiphenyl | 55 | 130 |
| TCMX | 70 | 125 |
| 8082 Water: | | |
| Decachlorobiphenyl | 40 | 135 |
| 8082 Solid: | | |
| Decachlorobiphenyl | 60 | 125 |

TABLE 3.4

HOLDING TIMES FOR LABORATORY WATER ANALYSES

| PARAMETER | METHOD | HOLDING TIME | CONTAINERS | PRESERVATIVE |
|----------------------|----------------------|--|-----------------------------------|---|
| TCLP | SW1311 | 14 days from collection to extraction, then follow liquid guidelines for each method using TCLP preparation as "collection" baseline | Four - 1 liter amber glass bottle | 4°C |
| TAL Metals | SW6010B | 6 months | One-1 liter plastic bottle | HNO ₃ , pH<2 |
| Mercury | SW7470A | 28 days | One-1 liter plastic bottle | HNO ₃ , pH<2 |
| Chromium, Hexavalent | SW7196A | 24 hours | 125ml plastic | 4°C |
| SVOCs | SW8270C | 7 days from collection to extraction; 40 days from extraction to analysis | Two-1 liter amber, glass bottle | 4°C |
| VOCs | SW5035A/8260B | 14 days | Three-40 ml, glass VOA vials | HCl, pH<2, 4°C |
| Pesticides | SW8081A | 7 days from collection to extraction; 40 days from extraction to analysis | One-1 liter amber, glass bottles | 4°C |
| TCLP Herbicides | SW8151A | 7 days from collection to extraction; 40 days from extraction to analysis | One-1 liter amber, glass bottle | 4°C |
| PCBs | SW8082 | 7 days from collection to extraction; 40 days from extraction to analysis | One-1 liter amber, glass bottle | 4°C |
| TPH | SW8015M | 14 days | One-1 liter glass bottle | HCL, 4°C |
| Extractable TOC | SW9060 Lloyd Kahn | 28 days | 100 mL, amber glass bottle | HCL or H ₂ SO ₄ , 4°C |
| Thorium | TH-NAS-3004 | 6 months | One-1 liter amber, glass bottle | HNO ₃ , pH<2, 4°C |
| Gross Alpha and Beta | SW9310 | 6 months | 500 mL, amber glass bottle | HNO ₃ , pH<2, 4°C |
| TSS | EPA 160.2 | 7 days | 1000 mL P/G bottle | 4°C |
| BOD-5 Day | EPA 405.1 | 48 hours | 1000 mL P/G bottle | 4°C |
| COD | EPA 410.4 | 28 days | 250 mL P/G bottle | 4°C, H ₂ SO ₄ to pH<2 |
| Cyanide | SW9012A | 14 days | One-1 liter plastic bottle | NaOH, pH>12, 4°C |

TABLE 3.4 (Continued)

HOLDING TIMES FOR LABORATORY WATER ANALYSES

| PARAMETER | METHOD | HOLDING TIME | CONTAINERS | PRESERVATIVE |
|-------------------------|-----------------|---------------------|----------------------------|---|
| pH | EPA 150.1 | immediate | 250 mL P/G bottle | 4°C |
| TKN | EPA 351.2 | 28 days | 500 mL P/G bottle | 4°C, H ₂ SO ₄ to pH<2 |
| Oil & Grease | EPA 413.1 | 28 days | 1000 mL glass bottle | 4°C, H ₂ SO ₄ to pH<2 |
| Oil & Grease | EPA 1664A | 28 days | 1000 mL glass bottle | 4°C, H ₂ SO ₄ to pH<2 |
| Nitrate as N | EPA 300.0 | 48 hours | 250 mL P/G bottle | 4°C |
| Nitrite as N | EPA 300.0 | 48 hours | 250 mL P/G bottle | 4°C |
| Total Organic Nitrogen | EPA 351.2/350.1 | 28 days | 500 mL P/G bottle | 4°C, H ₂ SO ₄ to pH<2 |
| Total Dissolved Solids | EPA 160.1 | 7 days | 500 mL P/G bottle | 4°C |
| Ammonia as N | EPA 350.1 | 28 days | 500 mL P/G bottle | 4°C, H ₂ SO ₄ to pH<2 |
| Total Hardness | SW6010B | 6 months | One-1 liter plastic bottle | HNO ₃ , pH<2 |
| Total Residual Chlorine | EPA 330.5 | immediate | 250 mL glass bottle | 4°C |
| Specific Conductance | EPA 120.1 | immediate | 250 mL glass bottle | 4°C |
| Temperature | EPA 170.1 | immediate | 250 mL glass bottle | 4°C |
| Turbidity | EPA 180.1 | immediate | 250 mL glass bottle | 4°C |
| Ignitability | SW1010/1020A | immediate | 125 mL glass bottle | 4°C |

P/G = plastic or glass bottle acceptable for this sample parameter

TABLE 3.5
HOLDING TIMES FOR LABORATORY SOIL ANALYSES

| PARAMETER | METHOD | HOLD TIME | CONTAINERS | PRESERVATIVE |
|----------------------|-----------------|---|---|--------------|
| TLCP | SW 1311 | 14 days from collection to extraction then follow solid guidelines for each method using TCLP preparation as the “collection” baseline. | 16 oz clear, wide-mouth glass jar | 4°C |
| TAL Metals | SW6010B | 6 months | 8 oz clear, wide-mouth glass jar | 4°C |
| Mercury | SW7471A | 28 days | 8 oz clear, wide-mouth glass jar | 4°C |
| Chromium, Hexavalent | SW3060A/SW7196A | 30 days from collection to digestion; 7 days from digestion to analysis | 8 oz clear, wide-mouth glass jar | 4°C |
| SVOCs | SW8270C | 14 days from collection to extraction; 40 days from extraction to analysis | 8 oz clear, wide-mouth glass jar | 4°C |
| VOCs | SW5035A/8260B | 48 hours from collection to preservation; 14 days from collection to analysis | Encore™ Sampler and 4oz clear, wide-mouth jar | 4 °C |
| Pesticides | SW8081A | 14 days from collection to extraction; 40 days from extraction to analysis | 8 oz clear, wide-mouth glass jar | 4°C |
| Herbicides | SW8151A | 14 days from collection to extraction; 40 days from extraction to analysis | 8 oz clear, wide-mouth glass jar | 4°C |
| PCBs | SW8082 | 14 days from collection to extraction; 40 days from extraction to analysis | 4 oz clear, wide-mouth glass jar | 4°C |
| TPH Extractable | SW8015M | 14 days | 4 oz clear, wide-mouth glass jar | 4°C |
| TOC | SW9060 | 28 days | 4 oz clear, wide-mouth glass jar | 4°C |
| | Lloyd Kahn | | | |
| Thorium | TH-NAS-3004 | 6 months | 4 oz clear, wide-mouth glass jar | 4°C |
| Gross Alpha and Beta | SW9310 | 6 months | 4 oz clear, wide-mouth glass jar | 4°C |

TABLE 3.5 (Continued)

HOLDING TIMES FOR LABORATORY SOIL ANALYSES

| PARAMETER | METHOD | HOLD TIME | CONTAINERS | PRESERVATIVE |
|------------------------|--------------------------|------------------|----------------------------------|---------------------|
| Corrosivity, pH | SW 9045C | Immediate | 4 oz clear, wide-mouth glass jar | 4°C |
| TKN | EPA 351.2 Modified | 28 days | 4 oz clear, wide-mouth glass jar | 4°C |
| Cyanide | SW9012A | 14 days | 4 oz clear, wide-mouth glass jar | 4°C |
| Nitrate as N | SW 9056 | 48 hours | 8 oz clear, wide-mouth glass jar | 4°C |
| Nitrite as N | SW 9056 | 48 hours | 8 oz clear, wide-mouth glass jar | 4°C |
| Total Organic Nitrogen | EPA 351.2/350.1 Modified | 28 days | 8 oz clear, wide-mouth glass jar | 4°C |
| Ammonia as N | EPA 350.1 Modified | 28 days | 8 oz clear, wide-mouth glass jar | 4°C |